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COMPLETE SPECIFICATION.

Detergent Tablets.

We, DETABGENT CORPORATION, a corporation organised and existing under the laws of the State of New York, United States of America, of Onondaga Street, Tully, New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to detersive preparations for use in washing and containing natural or synthetic detergents and particularly to a method for making detergent tablets and to the tablets so manufactured.

The tablets prepared by the process of the present invention are so controlled in composition and structure that they will not disintegrate in handling and tranportation, yet will readilv and rapidly disintegrate when in contact with water.

An important problem in the use of detergents in washing machines is convenient control of the amount of soap or detergent to be added to the washing water. It is difficult for the average person to judge and measure accurately the amount of detergent to be added for any particular load of material to be washed. There are advantages therefore in using a tabletted detergent made up in predetermined tablet units of detersive capacity. Also, tabletting of a detergent reduces the bulk volume by up to 80% for purposes of packaging, handling, transporting and storing.

The criteria for tabletted detergents are as follows:

1. The tablets must be strong and resistant to breakage and disintegration in the dry state.

2. They must rapidly disintegrate when in contact with water to release their active components.

3. They must be readily compressible to form tablets that will not stick to the walls of the die and punch faces used in their manufacture and the tablets must not laminate.

The use of tablets that dissolve and disintegrate rapidly in water has long been known in the pharmaceutical field, but neither the compositions needed to effect rapid disintegration nor the techniques used to make the tablets are feasible in producing a rapidly disintegrable tabletted detergent.

In tabletting pharmaceutical products the principal technique used for making tablets that disintegrate rapidly consists of including a small quantity of dry starch or similar substance in the material that is compressed in the mold. Upon contact with water the starch swells, breaking apart the tablet granules. Effervescence is also occasionally employed, the escaping gas serving to aid the disintegrating process. A bonding agent such as gelatin, glycerin, or other plastic material may also be used.

These techniques are not directly applicable to the manufacture of a rapidly disintegrating tabletted detergent, because of the nature of the detergents to be used. Effective detergents suitable for tabletting are generally of plastic nature and undergo substantial plastic flow when subjected to the pressures of the tabletting equipment while their characteristics as wetting agents causes them to stick to the punch faces and die walls. For instance, highly effective detergents such as the alkyl aryl sulfonates (e.g. dodecyl or tridecyl benzene sodium sulfonate), sodium lauryl sulfate, and the alkali salts of palmitic, stearic and oleic acids will not tablet success-

fully when subjected to tabletting techniques used in the pharmaceutical industry.

When these materials are compressed in a die to form tablets, even if mixed with substantial amounts of other solid granular or powdered materials, the tablets stick to the punch faces and die walls, producing broken tablets and stalling the tablet press. If sodium sulfate (a common filler in powdered detergents) or sodium chloride is added to give bulk, there is little or no improvement in compressibility.

According to the invention, a method for making a detergent tablet for washing purposes that disintegrates on contact with water comprises forming a concentrated solution or slurry of a solid organic surface-active agent in water, adding sodium chloride to the solution or slurry, removing the water from the solution or slurry, mixing the resulting dry material with an alkali carbonate and a solid hydrogen ion-producing material having an anion compatible with the detersive function of the composition, and compressing the mixture in a die to form tablets.

The alkali carbonate that is used is preferably sodium carbonate, sodium bicarbonate, or sodium sesquicarbonate ($Na_2CO_3.NaHCO_3.2H_2O$). The preferred hydrogen ion-producing component is a combination of sodium monophosphate (NaH_2PO_4) and boric acid. Sodium monophosphate alone is a desirable hydrogen ion-producing component, and others such as sodium bisulphate and boric acid that do not interfere with the detersive properties of the composition, may be used. The action of the preferred hydrogen ion-producing component provides CO_2 in reaction with the carbonate to accelerate the disintegration of the tablet, and the mixture of phosphate and borate ions constitutes a water softening component in the resulting solution. The proportions of the alkali carbonate and the hydrogen ion-producing component are varied to control the amount of CO_2 generated and thus the rate of disintegration of the tablet.

Besides these components it is also desirable to include starch (preferably cornstarch) or a conventional cellulosic disintegrating agent, and suitable inorganic detergent and water softening components, such as sodium tripolyphosphate, tetrasodium pyrophosphate, sodium metasilicate and sodium carboxymethyl cellulose. Other agents may be incorporated, such as sodium sulphate, sawdust, wood flour, silica gel and talc.

The preferred proportion of sodium chloride is from 10 to 50% and a proportion of 20 to 30% (based on the weight of detergent) is even more preferred.

The removal of water may readily be carried out by conventional spray-drying or other known techniques.

The following are examples of preferred compositions for tablets made according to the present invention. It will be understood that the compositions will vary depending upon the particular purpose for which the tablet is used. The ranges are expressed as % by weight.

TABLE I

2 to 50%	Alkyl aryl sodium sulfonate ($C_{12}H_{25}$ or $C_{13}H_{27}$)
0 to 45%	Sodium tripolyphosphate
0 to 10%	Sodium metasilicate
0 to 10%	Starch
10 to 45%	$NaHCO_3$
5 to 40%	NaH_2PO_4
0 to 20%	H_3BO_3
0 to 35%	Na_2SO_4
1 to 30%	NaCl

A preferred method of preparing the tablets of the present invention involves the following steps:

1. A 90% active wetting agent, such as sodium dodecyl or tridecyl benzenesulfonate is dissolved in hot water. The balance is sodium sulfate. Then to the solution is added an amount of sodium sulfate equal in weight to about one-third the weight of wetting agent added. Thereafter an amount of sodium chloride approximately equal to the added sodium sulfate is incorporated into the solution. The latter is then heated to evaporate water to such an extent that a slurry suitable for spray drying equipment is formed. The resulting slurry is then spray dried.

2. A dry mix of approximately 84% sodium metasilicate and 16% magnesium stearate is next prepared in a mechanical mixer. The purpose of the sodium silicate in the detergent is to prevent corrosion of metal surfaces in the washing equipment. However, this is difficultly compressible, even in the presence of sodium chloride; and tabletting is difficult, for this material tends to stick to the metal punch faces. However, by dry premixing the metasilicate with magnesium stearate, the latter coats the silicate and allows successful tabletting.

3. The dry solids obtained from steps 1 and 2 are then mixed in a mechanical mixer, along with ingredients previously enumerated in Table I. Furthermore, if desired, small amounts of dyes, perfumes and the neccessary alcohol to maintain these in solution may then be added in a suitable manner, as by spraying on the mix in the mechanical mixer.

The mix may then be dried at elevated temperatures to evaporate solvent, and then fed to the tabletting machine.

The final product obtained on solution in water has a pH in the range of about 7 to 9, depending upon the use to which it is adapted. The materials are so chosen and in such amounts that a 1 oz. tablet 2½" in diameter will disintegrate and be completely dissolved in 64 oz. of water at 60°C. in approximately 10 minutes. This is obtained, for instance, when 15% $NaHCO_3$ and 10% NaH_2PO_4 are present in the tablet. Increasing these reagents respectively to 35 and 50% causes solution in about 30 seconds. Thus by varying the amount of these components, release of detergent is effectively controlled.

The effect of sodium chloride, when added to a solution of wetting agent, may be illustrated by the following data:

WETTING AGENT: $C_{13}H_{27}.C_6H_4.SO_3Na$

% Wetting Agent	% Na_2SO_4	% NaCl	Compressibility	
			Before NaCl	After NaCl
45	55		Poor	
45	55	+25		Good
45	55	+50		Good
60	40		No	
60	40	+25		Fair
60	40	+50		Good
85	15		No	
85	15	+25		Fair
85	15	+50		Good

WETTING AGENT: $C_{12}H_{25}.C_6H_4.SO_3Na$

% Wetting Agent	% Na_2SO_4	% NaCl	Compressibility	
			Before NaCl	After NaCl
35	65		No	
35	65	25		Fair
35	65	50		Good
85	15		No	
85	15	25		Poor
85	15	50		Good
100	—		No	
100		25		Fair
100		50		Good

No: Material sticks to walls of die and punch faces so that first tablet is torn apart in removing from tool.
Poor: Only two or three tablets can be pressed before tablets are destroyed in removing from tool.
Fair: Three to ten tablets are compressed before difficulty begins.
Good: Over ten tablets with no difficulty.

From these results it is evident that the higher the detergent content the greater the amount of salt is necessary to provide the necessary compressibility. (As stated previously, however, the addition of equivalent amounts of salt to the detergent and the other components of the tablet in the dry state did not give a satisfactory product). Furthermore, sodium chloride added to a large number of commercial synthetic detergents in the dry state, containing also the phosphates, silicates and carbonates, did not give a compressible product.

The tablets of the present invention may be used for many purposes. They may be used for heavy detergent requirements, in bubble bath preparations, and the like. In Table II below are set forth some typical compositions which have been successfully tabletted and which have shown themselves highly effective for the purposes designated.

TABLE II

	Detergent	Bubble Bath	Milk Equipment Cleanser	Water Softener	Car Cleaner	Floor & Wall Cleaner	Cold Water Detergent
Alkyl benezene sodium sulfonate	13.50	13.50	9.0	2.25	9.0	6.75	27.0
Sodium sulfate	10.50	10.50	7.0	1.75	7.0	5.25	21.0
Sodium chloride	6	6	4.0	1.00	4.0	3.00	12.0
Sodium monophosphite ...	20	40	18.0	30.0	20.0	20	5.0
Sodium tripolyphosphate ...	20					15	10.0
Trisodium phosphate						15	
Tetrasodium pyrophosphate	5	5	20	10.0	20.0		
Sodium metasilicate	5						
Boric acid				10.0		5	
Sodium bicarbonate		10	10	15.0	15	10	
Sodium carbonate	9		10	20.0	15		5
Sodium sesquicarbonate ...		5	10			10	10
Starch (corn)	9	10	7.0	5.0	10	5	5
Talc				5.0		5	5
Magnesium stearate	1						
Sodium stearate			1.0				
Sodium carboxymethyl cellulose	1						
Dyes, perfumes, etc. ...	Less than 1%	Less than 1 %					Less than 1%
Sodium perborate			4.0				
	100	100	100	100	100	100	100

WHAT WE CLAIM IS:

1. A method for making a detergent tablet for washing purposes that disintegrates on contact with water, comprising forming a concentrated solution or slurry of a solid organic surface-active agent in water, adding sodium chloride to the solution or slurry, removing the water from the solution or slurry, mixing the resulting dry material with an alkali carbonate and a solid hydrogen

ion-producing material having an anion compatible with the detersive function of the composition and comprising the mixture in a die to form tablets.

2. A method as claimed in claim 1 in which the alkali carbonate is sodium carbonate, sodium bicarbonate or sodium sesquicarbonate.

3. A method as claimed in claim 1 or 2 in which the hydrogen ion-producing component is sodium monophosphate (NaH_2PO_4).

4. A method as claimed in claim 3 in which the hydrogen ion-producing component includes boric acid.

5. A method as claimed in any one of the preceding claims in which cornstarch is added to the said resulting dry material.

6. A method as claimed in any one of the preceding claims in which sodium tripolyphosphate, tetrasodium pyrophosphate, sodium metasilicate or sodium carboxymethyl cellulose is added to the said resulting dry material.

7. A method as claimed in any one of the preceding claims in which the proportion of sodium chloride is 10 to 50% based on the weight of the surface-active agent.

8. A method as claimed in claim 7 in which the proportion of sodium chloride is 20 to 30% by weight.

9. A method as claimed in any one of the preceding claims in which the surface-active agent is an alkyl aryl sodium sulfonate.

10. A method as claimed in any one of the preceding claims in which sodium sulphate is added to the slurry or solution of the surface-active agent.

11. A process as claimed in any one of the preceding claims in which a dry mixture of sodium metasilicate and magnesium stearate is added to the said resulting dry material.

12. A method as claimed in claim 1 substantially as described.

13. A detergent tablet whenever made by the process of any one of the preceding claims.

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